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DMIC

review OF RECENT DEVELOPMENTS

Beryllium

F. T. Zurey and S. H. Gelles • July 30, 1969

MECHANICAL METALLURGY

Mechanical Properties

An extensive program for developing test techniques to provide meaningful plastic property data for several beryllium-bearing materials over a broad temperature range has been conducted at the Lockheed Missiles & Space Company.(1) Elastic properties determined included Young's moduli in three directions, elastic stiffnesses, Poisson's ratios, and shear moduli at temperatures between 75 and 800 F. In addition, Young's modulus, elastic stiffness, and Poisson's ratios were determined at -320 F for hot-pressed beryllium block. Plastic properties (tensile properties, compressive yield strength, stress-strain curves, 3-point and 4-point bend tests) were also determined at temperatures between 75 and 800 F. The materials tested were PR-20 beryllium (powder sheet), S-200 beryllium (powder sheet), IS-Be ingot sheet, QMV high-strength beryllium (powder sheet), QMV hot-pressed beryllium (powder block), and Be-38Al (powder sheet in the annealed condition). Chemical compositions of the various materials were reported, but there was an absence of metallurgical structure and texture data. A summary of the data is presented in Table 1. Young's modulus for unalloyed beryllium decreased slightly (approximately 4 x 10^6 psi) with an increase in temperature from 75 to 800 F, in conflict with the anomalous increase in modulus reported by Salmen and Gobble. Poisson's ratio, however, remained constant as a function of temperature within the temperature range tested; elastic stiffness was almost constant, decreasing slightly. Biaxial stressing applied in the form of a 4-point bend test is of particular interest, and showed the following ranking of test materials based on permissible maximum fiber strain and minimum bend radius at 75 F.

	Minimum Bend Radius, inch	Maximum Fiber Strain, percent
Be-Al Alloy Sheet	<2[a]	>2.7[a]
Hot-Pressed Block	3	1.4
High-Strength Sheet	7	0.7
PR-20 Sheet	6	0.6
S-200 Sheet	7	0.5
Ingot Be Sheet	10	0.3

(a) No failure or cracks obtained.

The same ranking of materials was obtained from 3-point bend tests.

TABLE 1. SUMMARY OF ELASTIC PROPERTIES OF VARIOUS BERYLLIUM-BEARING MATERIALS(1)

Property	Direction	PR-20 Sheet	Ingot Sheet	Be-Al Alloy Sheet	High-Strength Sheet	Hot-Pressed Block	S-200[a] Sheet
			Test Temperature, 75 F				
E	L	41.7	40.1	28.6	42.4	--	43.3
E	T	42.7	42.7	30.1	--	42.6[b]	42.6
E	ST	51.4	48.9	33.2	50.2	--	51.5
G	L,T	--	19.6	...	--	-	19.9
G	L,ST	--	22.8	12.3	--	20.1[b]	21.3
TS	L	70	47	57	88	47[c]	78
TYS	L	50	39	40	75	36[c]	54
CYS	L	54	39	37	75	--	--
% Elong	L	7	2	11	3	NIL[c]	16
TS	T	78	53	49	99	56[b]	80
TYS	T	53	41	37	84	36[b]	56
CYS	T	53	40	35	82	--	--
% Elong	T	11	3	6	4	NIL	16
			Test Temperature, 400 F				
TS	L	53	39	39	70	45[c]	--
TYS	L	44	34	33	67	32[c]	--
% Elong	L	45	12	14	22	2.3[c]	---
TS	T	57	45	34	91	48[b]	--
TYS	T	46	36	30	76	34[b]	--
% Elong	T	58	33	8	37	14[b]	--
			Test Temperature, 800 F				
TS	L	31	26.3	20.3	55	39[c]	--
TYS	L	30	26.0	17.5	47.6	27[c]	--
% Elong	L	43	27.7	4.7	44.8	8[c]	--
TS	T	32	26.8	18.6	53.4	31[b]	--
TYS	T	31	26.2	16.3	48.6	24[b]	--
% Elong	T	54	40.4	2.8	25.0	36[b]	--

(a) Some of the data for S-200 were obtained in a previous study.
(b) Perpendicular to axis of pressing.
(c) Parallel to axis of pressing.
Note: E = modulus of elasticity; G = modulus of regidity; TS = ultimate tensile strength; TYS = tensile yield strength; CYS = compressive yield strength; Elong = elongation; L = longitudinal; T = transverse; ST = short transverse

The results of an experimental investigation to determine the room-temperature mechanical properties of commercially produced Be-38 Al alloy tubing (Lockalloy) have been reported by Langley Research Center.(2) Tubes ranging in diameter from 0.25 to 0.69 inch with wall thicknesses of 0.020 inch were tested in both the as-extruded and annealed conditions. Along with tensile and compressive mechanical property determinations, tests were conducted on the column behavior and microhardness and metallurgical examinations were carried out at both a macroscopic and microscopic level. The results of detailed dimensional measurements indicated that Be-38 Al alloy tubing can be produced commercially to dimensional tolerances comparable with those of aluminum tubing. A summary of the mechanical properties of extruded tubing is given in Table 2.

Microplastic Behavior

The microplastic characteristics of hot isostatically pressed and isoforged beryllium were

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TABLE 2. SUMMARY OF ROOM-TEMPERATURE MECHANICAL PROPERTIES OF EXTRUDED Be-38 Al ALLOY TUBING(2)

Outside Diameter, inches	0.2% Offset Tensile Yield Strength, ksi	Ultimate Tensile Strength, ksi	0.2% Offset Compressive Yield Strength, ksi	Modulus of Elasticity, 10^6 psi	Elongation in 2 Inches, percent
		As Extruded			
[illegible]	[illegible]	66.2	71.2	28.9	< 1
0.4375	--	65.0	74.7	28.7	< 1
0.5625	78.[illegible]	81.3	76.9	28.6	< 1
0.6875	81.[illegible]	85.6	78.0	29.0	< 1
		Annealed			
0.[illegible]00	50.[illegible]	56.8	55.7	28.7	2
0.4375	50.[illegible]	53.4	48.6	29.1	2
0.5625	48.[illegible]	60.3	43.8	30.4	5
0.6875	[illegible]6	54.5	43.4	29.3	3

studied by Shemenski and Maringer.(3) By comparison, hot isostatic pressing typically makes use of hot-gas pressure acting upon a readily deformable evacuated container while isoforging employs a solid pressure transmitting medium, such as steel or alumina, enclosed in a cavity and acted on by the ram of a forging press. Typical pressing parameters for hot isostatic pressing were 1675 F and 10 ksi for 2 hours, while isoforging employed greater pressures, from 30 to 145 ksi, for shorter times, from 12 to 120 seconds. Both processes employed significantly higher pressures and lower temperatures than are normally employed for commercially compacted beryllium.

The flow stress of isostatically pressed beryllium was found to be a parabolic function of microstrain. The flow curves were characterized by the following three distinct ranges of microstrain: (1) heterogeneous basal glide at low microstrain, (2) hardening, most probably by a dislocation pile-up mechanism at intermediate levels of microstrain, and (3) prismatic glide and cross-slip at microstrains greater than 50×10^{-6}. Microflow behavior was found to be a sensitive function of fabrication history as illustrated in Figure 1. The microyield (10^{-6} offset yield) strength is observed to increase with decreasing processing temperature, increasing beryllium oxide content, and small alloying additions of chromium or silver. The highest microyield strength observed was 103.5 ksi for an alloy containing 0.27 weight percent chromium.

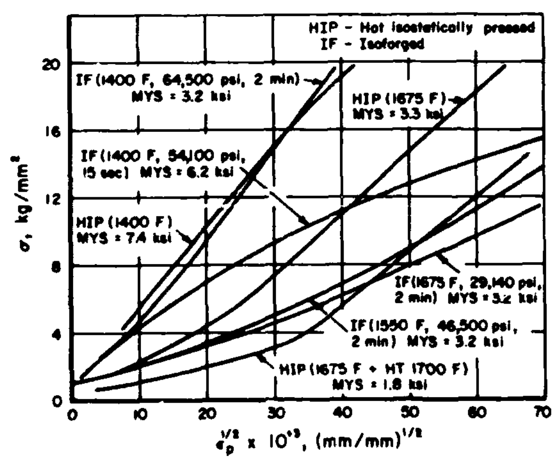


FIGURE 1. VARIATION OF PARABOLIC MICROSTRAIN BEHAVIOR AS A FUNCTION OF FABRICATION HISTORY FOR AS-RECEIVED S-200 GRADE BERYLLIUM(3)

Grain-Size Hardness Relationship

The hardness dependence on grain size of high-purity polycrystalline beryllium produced from SR-grade electrolytic flake melted in high vacuum in an electron-beam furnace has been determined by Bunshah and Armstrong.(4) The principal impurities in the metal in ppm atomic were as follows: 30 oxygen, 10 carbon, 5 aluminum, 4 chlorine, 7 iron, and 2 nickel. All other impurities were 1 ppm or less.

The hardness was found to agree with the following relation:

$$H = H_0 + K_H \ell^{-1/2}$$

where ℓ is the grain diameter and H_0 and K_H are constants. This relation is similar to the one for yield strength versus grain size,

$$\sigma_y = \sigma_{oy} + K_y \ell^{-1/2}.$$

The hardness and yield strength of higher purity material was significantly lower than values determined by Macres on relatively impure extruded beryllium rod: Armstrong and Bunshah have used Macres' data to compute the constants, H_0, K_H (Meyer Hardness Number), and σ_{oy} and K_y. The constants determined are presented in Table 3.

TABLE 3. HARDNESS AND YIELD STRENGTH GRAIN-SIZE CONSTANTS(4)

H_0 (Kg/mm^2)	K_H(Kg/mm$^{3/2}$)	σ_{oy} (Kg/mm^2)	K_y(Kg/mm$^{3/2}$)
150	2.7	17.5	0.5

PHYSICAL METALLURGY

Self-Diffusion

Self-diffusion in polycrystalline beryllium by using the ^{7}Be isotope has been investigated by the Russians.(5) The specific activity was plotted as a function of the square of the diffusion distance for temperatures of 650, 800, 900, 1000, and 1200 C (1200 to 2190 F). The resultant experimental values of the diffusion coefficient are presented in Figure 2. The diffusion coefficient as a function of temperature was calculated to be:

$$D = 0.36 \exp\left(-\frac{38400}{RT}\right) cm^2/sec.$$

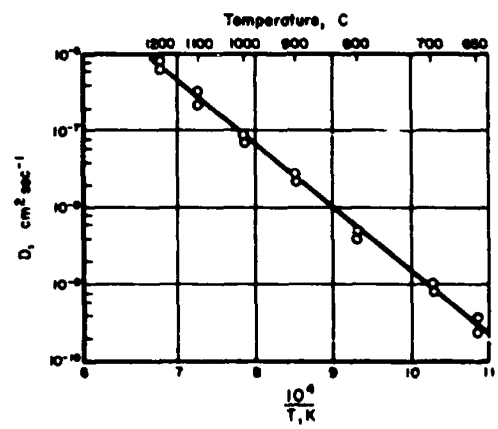


FIGURE 2. DIFFUSION COEFFICIENT FOR SELF-DIFFUSION IN POLYCRYSTALLINE BERYLLIUM OVER THE TEMPERATURE RANGE 650 to 1200 C(5)

Substructure

A novel technique for identifying anisotropic effects and inhomogeneities of defect structures has been presented by Glass and Weissman.(6) The technique involves the combined analysis of deficiency conics produced by divergent X-ray techniques and X-ray topographs. The method has been applied to the room-temperature deformation of beryllium.

Annealed single crystals were found to contain subgrains, columnar in shape, having dimensions of the order of 1 millimeter along the c-axis and a cross section of about 50 microns in diameter. The misorientation between neighboring subgrains was in the order of 1 or 2 minutes of arc. Also, the topographs revealed the existence of a finer substructure leading to an additional orientation spread of 1 minute of arc. Basal and prismatic changes in response to compressive deformation were found to be inhomogeneous because of variations in the defect structure initially present.

Russian Literature Review

A book on the physical metallurgy of beryllium has been published by the Russians.(7) The book is intended for scientists, engineers, and technicians working in the field of the metallurgy of reactor materials. Methods used to produce high-purity beryllium, primarily methods such as vacuum melting, zone melting, and refining, are reviewed. Various physical properties of beryllium metal (eg., electrical, thermal, magnetic, elastic, internal friction, and diffusion), its crystal structure, thermodynamic characteristics, and solidification features are described. A great deal of attention is devoted to the consideration of the influence of such factors as composition, structure, and heat treatment on the physical properties of beryllium. Data on the effect of neutron and α radiation upon the structure and physical and mechanical properties of beryllium are given. New methods in beryllium research, such as electron microscopy, electron diffraction, high-temperature X-ray analysis, and microbeam probe analysis of specimens, are also described. The book, in Russian, contains 75 tables, 129 figures, and bibliography. Of the total 813 references, only 140 are Soviet.

Another Russian reference, that contains essentially the same information as the one above, is the Encyclopedia of Modern Engineering, Structural Materials (Sovetskaya Ensiklopediya, Moscow, 1964). Recently, DMIC obtained translated extracts from this reference and published them as DMIC Technical Note, "Extracts on Soviet Beryllium and Beryllium Alloys", June 1969. A copy of the technical note is available on request.

POWDER METALLURGY

Hot Isostatic Pressing

A study to develop the hot isostatic pressing (HIP) process for the production of powder-metallurgy beryllium equivalent or superior to "S-200-type" vacuum-hot-pressed block was conducted at Battelle's Columbus Laboratories.(8)

In the hot isostatic pressing process, powder of commercial purity, with about 1 weight percent oxide and of -220 mesh particle size, was used in the program. The beryllium powder is first loaded into a shaped latex bag. A vacuum is then drawn on the latex bag, and it is sealed. External atmospheric pressure on the assembly provides sufficient rigidity for handling. It is then cold hydrostatically pressed to pressures of 30 to 100 ksi, which produces a green pressing with sufficient strength for ease of handling. The green pressing is fitted and sealed in a steel pressing container. Both mild steel and Type 304 stainless steel are suitable pressing container materials. The green pressing is then outgassed and the outgassing stem sealed by forging and welding. Typically, outgassing is achieved at 1200 F for 20 to 24 hours. The outgassing assembly is compressed by hot isostatic pressing in a hot-gas autoclave. Pressing is typically performed at 1675 F and 10 ksi for 2 hours. Finally, the HIP container is removed and the beryllium specimen is ready for any necessary machining operations.

During the program, the variable parameters of outgassing treatment, pressing temperature, annealing temperature, powder size fraction, and powder grade were studied in terms of their effect on microstructure, density, and room-temperature tensile properties of HIP beryllium.

Some of the more significant observations of the study are summarized as follows:

(1) Fully densified beryllium that had been insufficiently outgassed had little ductility and swelled during annealing.

(2) Optimum tensile properties, as controlled by the outgassing procedure, probably would be obtained by imparting a controlled amount of contamination to the powder for strengthening while removing the volatile constituents.

(3) Approximately 0.1 weight percent volatiles (as H_2O, CO_2, and CO) were found to be present in beryllium powder prior to outgassing. Also, once outgassed, the powder acts as a getter to become recontaminated.

(4) Powder-size variations did not affect swelling during post-pressed annealing of beryllium.

(5) The minimum HIP temperature to achieve full densification was found to be 1300 F at 10 ksi for 2 to 3 hours.

(6) The microstructure of beryllium densified at 1400 F contains a network unresolved by optical microscopy with fine-grained areas along it. This network has been associated with the initial powder particle surfaces. With increased pressing or annealing temperature, the amount of fine microstructure decreased.

(7) As the pressing or annealing temperature was increased, the room-temperature elongation increased with a corresponding decrease in tensile ultimate and yield strength.

(8) The general effect of powder size on tensile properties was to increase yield strength and decrease ductility as powder size was decreased.

Figure 3 illustrates the effect of annealing temperature on the tensile properties of HIP beryllium.

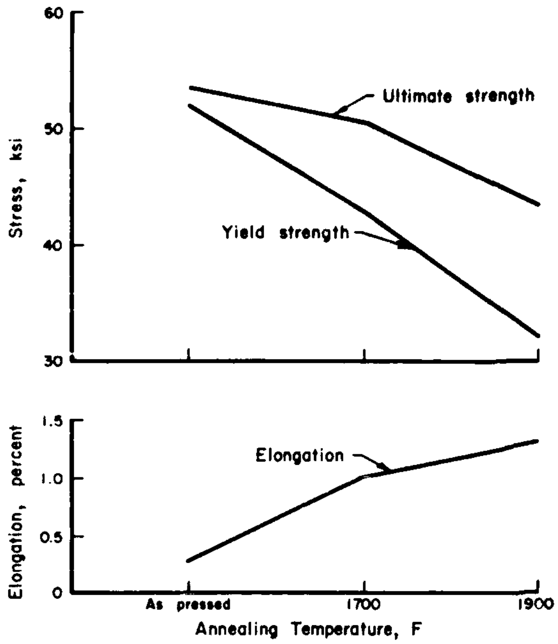


FIGURE 3. EFFECT OF ANNEALING TEMPERATURE ON THE TENSILE PROPERTIES OF BERYLLIUM HOT ISOSTATICALLY PRESSED AT 1400 F(8)

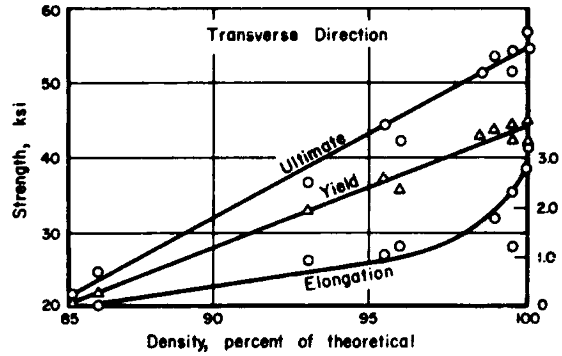


FIGURE 4. ROOM-TEMPERATURE TENSILE PROPERTIES AT VARIOUS DENSITIES(10)

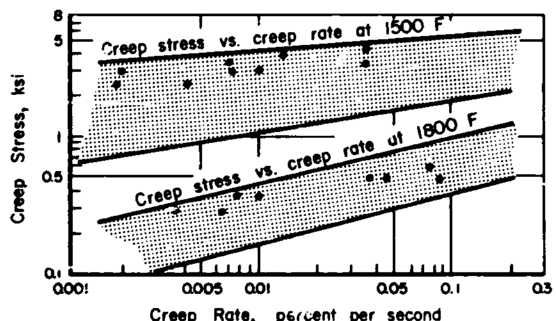


FIGURE 5. COMPRESSION CREEP PROPERTIES(10)

Submicron Powder

An investigation of a process for producing submicron beryllium powder and fine-grained beryllium metal was conducted by the National Research Corporation.(9) The major consideration during consolidating submicron powder into solid metal was to maintain an extremely fine grain size. Although the process was proven feasible for producing very fine-grained beryllium, inconclusive results were obtained in defining the role of submicron grain size on the mechanical properties.

The submicron beryllium powder was produced by evaporation of beryllium metal in an argon atmosphere ranging from about 5 to 300 μ. The evaporated beryllium vapor was collected on a water-cooled, rotating stainless steel drum which had been given an adherent beryllium coating to reduce contamination. The oxide content of the resultant powder was found to be dependent on the argon pressure. The beryllium powder produced by this method consisted of black spherical agglomerates with an individual particle size of less than 0.1 μ. This powder was free flowing and pyrophoric.

Two techniques were employed to consolidate the powder. The first was to cold press and then sinter; the second was to cold press and then hot isostatically press. A maximum sintering temperature of 1550 F was found to produce a material without excessive grain growth but with a density of only about 70 percent. Hot isostatic pressing the powder at 1400 F at 10 ksi for 2 hours resulted in a beryllium pressed density of about 99 percent and a grain size of the order of a micron. Failure to produce suitable material for evaluating mechanical properties was due largely to unidentified powder contamination during processing and to nonoptimum processing employed in the hot isostatic pressing.

Spark Sintering

The spark sintering process for the fabrication of powder-metallurgy beryllium structures has been developed by Lockheed.(10) In this process, sintering is accomplished by an electrical discharge through the powder while it is held under pressure in a die. Dimensions of the largest billets presently being fabricated are 7-1/2-inch-diameter by 10-inches high. Billets with densities as low as 65 percent and as high as 100 percent of theoretical can be produced by this method. Typical room-temperature tensile properties of 100 percent dense, spark-sintered beryllium tested longitudinally are as follows: 48 ksi ultimate tensile strength, 38 ksi 0.2 offset yield strength, and 1.4 percent elongation. The tensile properties as a function of density and the elevated-temperature creep properties of the spark-sintered beryllium are presented in Figures 4 and 5.

CORROSION

The oxidation of (1) sintered beryllium materials with various oxide contents, (2) a Be-5Ca alloy, and (3) the beryllide $CaBe_{13}$ has been studied by the Russians.(11,12) The corrosion environment employed was a free stream of carbon dioxide at 700 C (1290 F) containing 0.0005, 0.2, or 12 volume percent water vapor for exposures of up to 1000 hours. All the materials tested in the dried carbon dioxide (0.0005 volume percent water vapor) proved to be corrosion resistant. Raising the moisture content increased the oxidation rate considerably. Greater corrosion resistance was observed for the beryllide,

the Be-5Ca alloy, and the higher oxide sintered beryllium. The corrosion rates of the various beryllium alloys in carbon dioxide at the different water-vapor contents are presented in Figure 6. Some of the higher oxide beryllium was prepared by preoxidizing the powders. The distribution of beryllium oxide particles on the powder was of considerable importance. Uniform and controlled oxidation of the powder and a fairly uniform distribution of the oxides along the grain boundaries in the compacted beryllium was obtained by oxidizing the powder in a rotating furnace at 600 to 800 C with an atmosphere of argon plus oxygen. This material with the uniform oxide distribution had substantially improved corrosion resistance. The corrosion resistance was also dependent on the prior treatment of the specimens. Preoxidation in dry carbon dioxide increased the corrosion resistance in humid carbon dioxide. Annealing in a humid environment decreased the corrosion resistance. This was attributed to hydrogen pickup. The original corrosion resistance could be restored by vacuum annealing. Also hydrogen analysis of specimens corrosion tested at 700 C in CO_2 with 0.2 volume percent H_2O showed that the more corrosion-resistant material had lower hydrogen content. Therefore, it was concluded that the low corrosion resistance of beryllium in humid atmospheres is mainly due to hydrogen adsorption.

Water-vapor corrosion (5 and 30 mm Hg) at temperatures from 600 to 800 C (1110 to 1470 F) was studied by the Japanese with the use of a Gulbransen-type microbalance.[13] At 600 C, the oxidation rate followed the parabolic rate law during the initial period of oxidation, after which it conformed to a logarithmic pattern. At 650 and 700 C, the oxidation proceeded first by a logarithmic model which then changed to follow a rectilinear rate law. The latter step was accompanied by spalling of the oxide film. At 800 C, the oxidation obeyed the rectilinear rate law throughout the course of the experiment. Electron metallography indicated that the logarithmic rate law can be attributed to the generation of blisters at the oxide-metal interface and that the ensuing rectilinear pattern is introduced by spalling of the oxide film.

Chemical conversion coatings on beryllium for corrosion protection were studied by Booker and Stonehouse.[14] Coatings of chromate, phosphate-chromate, oxalate, and phosphate were applied by dipping specimens in commercial solutions. Corrosion conditions tested were high-humidity air at 170 F, salt spray, and high-temperature oxygen at 1290 to 1700 F. The chromate coatings were found to provide the greatest protection. The phosphate and oxalate coatings did not impart suitable corrosion resistance to the beryllium.

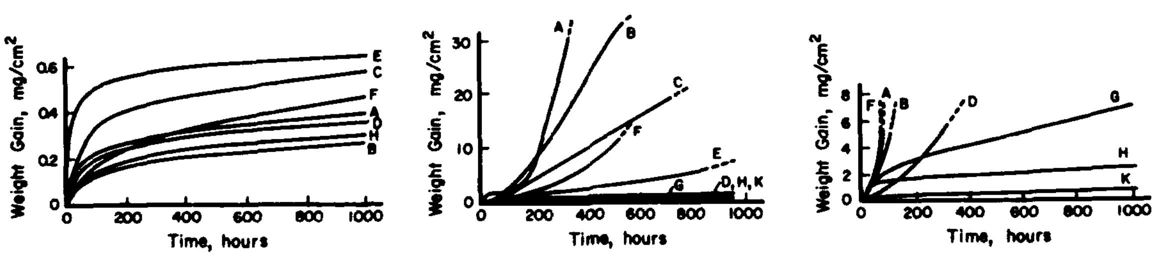
NEW PROGRAMS

Fabrication

"Production Techniques for Beryllium Wire", United Aircraft Corporation, Contract N00019-69-C-0236.

"Develop Beryllium Wire-Metal Matrix Composites for Use in Gas Turbine Fans and Compressors", Allison Division, General Motors Corporation, Contract N00019-69-C-0234.

Material Designation	Particle Size of Starting Powder, μ	BeO, weight percent	Notes
A	-500	0.1-0.3	
B	-50	0.8-1.3	Grinding of condensed material in a ball mill
C	-50	0.1-0.2	Grinding of condensed material in a disk attrition mill
D	-50	3.5	Additional oxidation of powder C in an Ar + O_2 mixture
E	-50	5.5	Additional oxidation of powder B in air without agitation
F	--	--	Vacuum-fused condensed material
G	≤1	10-12	Obtained by arc atomization in an inert atmosphere
H	Be-50 Ca-160	1-1.5	Beryllium alloy with 5 percent calcium
K	Be-50 Ca-160	1-1.5	Beryllide $CaBe_{13}$



(a) Oxidation kinetics of sintered beryllium materials in dried carbon dioxide at 700 C. H_2O content ≤0.0005 vol %.

(b) Oxidation kinetics of sintered beryllium materials in carbon dioxide with 0.2 vol % H_2O at 700 C.

(c) Oxidation kinetics of sintered beryllium materials in carbon dioxide with 12 vol % H_2O at 700 C.

FIGURE 6. WEIGHT-GAIN MEASUREMENTS OF THE SINTERED BERYLLIUM ALLOYS[11]

Alloying

"Mechanisms of Inhibiting Crack Growth and Effects of Alloying on the Electronic Structure of Beryllium", The Franklin Institute Research Laboratories, Contract F33615-67-C-1367.

"A Testing Program to Develop Improved Beryllium Alloys", The Franklin Institute Research Laboratories, Contract N00019-69-C-0250.

Mechanical Properties

"Mechanical Properties of Beryllium at High Strain Rates", General Motors Technical Center.

Miscellaneous

"Beryllium Research Materials", The Brush Beryllium Company, Contract F33616-69-C-1168.

"The Deposition of Aluminum Alloys and Beryllium", Electro Optical Systems Division, Xerox Corporation, Contract NAS 1-8953.

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(5) Pavlinov, L. V., Grigor'ev, G. V., and Sevast'yanov, Yu. G., "Beryllium Self Diffusion", Fizika Metallov i Metallovedeniya, 25 (3), 565-567 (March 1968).

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(10) "Spark-Sintered Beryllium", Data Sheet, Lockheed Missiles & Space Company, Sunnyvale, Calif. (March 1, 1969).

(11) Ivanov, V. E., et al. "Sintered Beryllium-Base Compositions-II. Corrosion Properties and Possibilities of Raising Corrosion Resistance in Humid Atmospheres", J. Soviet Powder Metallurgy & Metal Ceramics, 8 (68), 647-650 (August 1968).

(12) Ivanov, V. E., et al, "Sintered Beryllium-Base Compositions-I. Preparation and Some Mechanical and Thermophysical Properties", J. Soviet Powder Metallurgy & Metal Ceramics, 7 (67), 534-536 (July 1968).

(13) Sasabe, M., and Nomura, S., "High-Temperature Oxidation of Beryllium in Water Vapor", Nippon Genshiryoku Gakkaishi, 10, 319-324 (1968).

(14) Booker, J., and Stonehouse, A. J., "Chemical Conversion Coatings Retard Corrosion of Beryllium", Materials Protection, 8 (2), 43-47 (February 1969).

DMIC Reviews of Recent Developments present brief summaries of information which has become available to DMIC in the preceding period (usually 3 months), in each of several categories. DMIC does not intend that these reviews be made a part of the permanent technical literature. Copies of referenced reports are not available from DMIC; most can be obtained from the Defense Documentation Center, Cameron Station, Alexandria, Virginia 22314.

R. W. Endebrock, Editor